

# PATENT ABSTRACTS OF JAPAN

(11)Publication number : 09-158710

(43)Date of publication of application : 17.06.1997

(51)Int.Cl.

F01N 3/02  
B01D 39/00  
B01D 39/14  
B01D 39/20  
B01D 46/00  
B01D 53/94  
B01J 37/02

(21)Application number : 07-320107

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(22)Date of filing : 08.12.1995

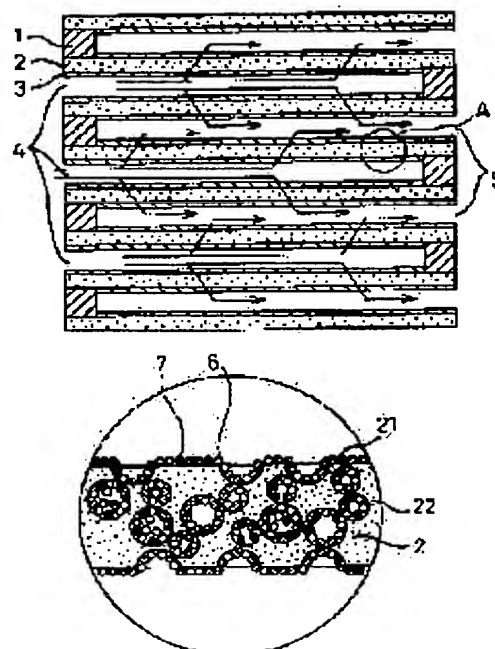
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## (54) DIESEL EXHAUST GAS PURIFYING FILTER

(57)Abstract:

**PROBLEM TO BE SOLVED:** To increase the contact area of exhaust gas with a coating layer so as to improve reactivity to an oxidation catalyst and thereby to improve purifying performance by providing a structure body with coating material adhering to the surface and the inside of pores of a cell side wall of a honeycomb type filter.

**SOLUTION:** In a porous ceramic filter of honeycomb structure, both ends of monolithic honeycomb are alternately plugged with plugging material 1. Activated alumina coating layers 3 are formed on the surface 21 of a cell side wall 2 of a honeycomb type filter and the pore surface 22 of the cell side wall 2. Diesel exhaust gas containing particulates enters into a cell from the cell inlet side 4, passes through the cell side wall 2 and goes out from the cell outlet side 5. At this time, the particulates are collected by surface and internal pores of the cell side wall 2. The filter thus coated can be suitably used as a diesel particulate filter with a low pressure loss.



## LEGAL STATUS

[Date of request for examination]

28.03.2002

[Date of sending the examiner's decision of rejection]

[Kind of final disposal of application other than the examiner's decision of rejection or application converted registration]

[Date of final disposal for application]

[Patent number]

3707843

[Date of registration]

12.08.2005

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CLAIMS

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[Claim(s)]

[Claim 1] One end of cel opening of a porosity ceramic honeycomb structure object which has an open cell every other piece \*\*\*\*\*, Cel opening to which \*\*\*\*\* is not carried out at this edge the edge of the opposite side A \*\*\*\* suggestion \*\*\*\* cage, It is the honeycomb mold filter with which the pore in a cell wall is passed and gas circulates. The diesel emission-gas-purification filter with which the pore in the cell wall which has the structure in which the coating ingredient containing a high specific-surface-area ingredient particle has adhered to the interior of the front face of a cel side attachment wall and the pore of a cel side attachment wall is passed, and gas circulates.

[Claim 2] The filter according to claim 1 whose average pole diameter the porosity of the cell wall of the filter after supporting said high specific-surface-area ingredient is 5-35 micrometers at 40 - 65%.

[Claim 3] The diesel emission-gas-purification filter according to claim 1 or 2 said whose high specific-surface-area ingredient is an activated alumina.

[Claim 4] Furthermore, a filter given in any 1 term of claims 1-3 with which the catalyst metal which consists of at least one sort of platinum group metals is supported.

[Claim 5] The porosity ceramic honeycomb structure object which has an open cell is coated with the coating slurry containing a high specific-surface-area ingredient particle and an inflammable destruction-by-fire matter particle. Under the present circumstances, the thing which the mean particle diameter of said high specific-surface-area ingredient particle and an inflammable destruction-by-fire matter particle is magnitude smaller than the average pole diameter of said honeycomb structure object, and is calcinated after that, Opening which \*\*\*\*\* one end of cel opening of said honeycomb structure object every other piece in a list, and is not \*\*\*\*\* (ing) at this edge is the manufacture approach of a diesel emission-gas-purification filter that pass the pore in a cell wall including \*\*\*\*\* (ing) the edge of the opposite side, and gas circulates.

[Claim 6] Said inflammable destruction-by-fire matter particle is carbon, and said coating slurry is the manufacture approach of claim 5 of said high specific-surface-area ingredient particle which contains said 5wt(s)% carbon particle at least.

[Claim 7] Said honeycomb structure object is coated with the slurry which does not contain a high specific-surface-area ingredient including an inflammable destruction-by-fire matter particle beforehand, and it is made to dry, coats with the slurry which does not contain the inflammable destruction-by-fire matter including a quantity specific-surface-area ingredient particle after that, and calcinates, Opening which \*\*\*\*\* one end of cel opening of said honeycomb structure object every other piece in a list, and is not \*\*\*\*\* (ing) at this edge is the manufacture approach of a filter that pass the pore in a cell wall including \*\*\*\*\* (ing) the edge of the opposite side, and gas circulates.

[Claim 8] The manufacture approach according to claim 7 that the percentage that the inflammable destruction-by-fire matter particle in the slurry which does not contain a high specific-surface-area ingredient particle including said inflammable destruction-by-fire matter particle occupies is 5 - 50wt%.

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**DETAILED DESCRIPTION**

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**[Detailed Description of the Invention]****[0001]**

**[Field of the Invention]** This invention removes a particulate at least among the matter contained in the gas discharged by internal combustion engines, such as a diesel power plant, and relates to the filter for PATI queue rate uptake used in order to purify exhaust gas.

**[0002]**

**[Description of the Prior Art]** The matter harmful to the body is contained in the particulate discharged by internal combustion engines, such as a diesel power plant, and it has been a technical problem on an environment to remove this. For this reason, in the former, after it carries out uptake of the particulate and it carries out constant-rate uptake with the filter prepared in the exhaust air system of a diesel power plant, the approach of carrying out combustion removal of the particulate by the electric heater, a burner, etc. is performed. Moreover, the combustion temperature of a particulate [ the platinum metal catalyst supported in the filter ] is lowered, and there is also a method of burning continuously the particulate which carried out uptake. In the case of the approach of carrying out combustion removal of the particulate in which the former carried out uptake by the electric heater, a burner, etc., the filter maximum temperature at the time of combustion rises, a filter may be damaged with the thermal stress concerning a filter, and the particulate amount control of uptake is so important that there are many particulate amounts of uptake, but it has come to control the amount of uptake completely. Since the thermal stress which combustion temperature becomes comparatively low and is applied to a filter becomes small in combustion by the latter catalyst, a catalyst is excellent in thermal resistance.

**[0003]** Generally in the above-mentioned approach, the cordierite which shows low-fever expansibility is mainly used for particulate uptake as the quality of the material, using the honeycomb structure object of a ceramic in many cases.

**[0004]** Cel opening of one end of the ceramic monolith of honeycomb structure, for example, cel opening by the side of a gas inlet, is \*\*\*\*\* (ed) for the diesel emission-gas-purification filter with which this invention is related every piece, and cel opening by the side of a gas outlet is chisel \*\*\*\*\*(ed) about the cel which opening of an entrance side is not \*\*\*\*\* (ing). Therefore, exhaust gas passes the pore of a cel side attachment wall, and uptake of the particulate which flows with exhaust gas is carried out inside the front face of this cel side attachment wall, and the pore of a cel side attachment wall.

**[0005]**

**[Problem(s) to be Solved by the Invention]** When the honeycomb structure porosity ceramic filter for diesel exhaust gas \*\*\*\*\* the both ends of a monolith honeycomb by turns as mentioned above, gas has an advantage with particulate collection efficiency higher than the filter of other structures for the structure of flowing in the cel which passes the several micrometers - dozens of micrometers pore of a cell wall, and adjoins. The above \*\*\*\*\* are made and this invention offers the diesel emission-gas-purification filter which has the porosity ceramic filter by which coating was carried out by high specific-surface-area ingredient particles, such as an activated alumina, in the interior of a cel side-attachment-wall front face and its pore.

**[0006]** When coating the ceramic support of honeycomb structure with high specific-surface-area ingredient particles, such as an activated alumina, what the above-mentioned combustible material is made burned down, forms much pores in the front face of a coating layer, and raised the diffuser efficiency of the gas to a coating layer by this pore is well-known by adding combustible material particles, such as carbon, to the slurry of an activated alumina, coating a carrier surface with this slurry and calcinating it (JP,57-99314,A, JP,61-245849,A). However, this aims at increasing the touch area to the coating layer of exhaust gas, raising

reactivity with an oxidation catalyst about the ceramic honeycomb of a flow through type, and raising the purification engine performance. That is, the exhaust gas with which it is the purpose increasing the surface area of a coat layer by building a depression with about about 10-20-micrometer pore with carbon on the front face of the coating layer of an activated alumina and with which this invention is related differs from the diesel emission-gas-purification filter which passes a cel side attachment wall fundamentally.

[0007]

[Means for Solving the Problem] One end of cel opening of a porosity ceramic honeycomb structure object which has an open cell the 1st mode of this invention every other piece \*\*\*\*\*, Cel opening to which \*\*\*\*\* is not carried out at this edge the edge of the opposite side A \*\*\*\* suggestion \*\*\*\* cage, It is the honeycomb mold filter with which the pore in a cell wall is passed and gas circulates. It is the diesel emission-gas-purification filter with which the pore in the cell wall which has the structure in which the coating ingredient containing a high specific-surface-area ingredient particle (only henceforth a "high specific-surface-area ingredient") has adhered to the interior of the front face of a cel side attachment wall and the pore of a cel side attachment wall is passed, and gas circulates.

[0008] The 2nd mode of this invention the porosity ceramic honeycomb structure object which has an open cell It coats with the coating slurry which contains an inflammable destruction-by-fire matter particle (only henceforth "the inflammable destruction-by-fire matter") with a high specific-surface-area ingredient. Under the present circumstances, the thing which the mean particle diameter of said high specific-surface-area ingredient and the inflammable destruction-by-fire matter is magnitude smaller than the average pole diameter of said honeycomb structure object, and is calcinated after that, Opening which \*\*\*\*\* one end of cel opening of said honeycomb structure object every other piece in a list, and is not \*\*\*\*\* (ing) at this edge is the manufacture approach of a diesel emission-gas-purification filter that pass the pore in a cell wall including \*\*\*\*\* (ing) the edge of the opposite side, and gas circulates.

[0009] As for the porosity ceramic honeycomb structure object used for this invention, it is desirable to be made from the cordierite (chemical composition type  $2\text{MgO}$ ,  $2\text{aluminum}_2\text{O}_3$ , and  $5\text{SiO}_2$ ) conventionally known as low-fever expansibility ceramics. The porosity of this can be freely adjusted by the well-known approach to this contractor.

[0010] What is necessary is to coat said porosity ceramic honeycomb structure object with the coating ingredient and the coating slurry which contains the inflammable destruction-by-fire matter preferably containing a high specific-surface-area ingredient, and to remove an excessive slurry desirable after that, and just to calcinate, in order to make the coating ingredient which contains a high specific-surface-area ingredient for said high specific-surface-area ingredient inside the front face of the cel side attachment wall of said porosity ceramic honeycomb structure object, and the pore of a cel side attachment wall adhere.

[0011] Usually, after making said porosity ceramic honeycomb structure object support high specific-surface-area ingredients, such as an activated alumina, the catalyst metal which was excellent in the engine performance which burns the particulate in exhaust gas by making this high specific-surface-area ingredient into support is made to support, the above-mentioned \*\*\*\* is carried out, and a diesel emission-gas-purification filter is done. The amounts of the catalyst metal in this case will be few, and the porosity of the above-mentioned diesel exhaust gas filter will become settled mostly in the porosity of the phase where the above-mentioned quantity specific-surface-area ingredient was made to support. 40 - 65% of the porosity of the cell wall of the honeycomb structure object in the phase where said high specific-surface-area ingredient was made to support is desirable, and is still more desirable. [ 45 - 60% of ] Moreover, the average pole diameter of this cell wall has desirable 5-35 micrometers, and its 10-30 micrometers are still more desirable. If this porosity is [ an average pole diameter ] 5 micrometers or more at 40% or more, pressure loss in case exhaust gas passes this filter will be small, and an engine output will not decline. On the other hand, at 65% or less, an average pole diameter will not become [ particulate capture ability ] so low, if said porosity is 30 micrometers or less.

[0012] Said high specific-surface-area ingredient is high specific surface area, so that the mean particle diameter is smaller than the average pole diameter of said porosity ceramic honeycomb structure object, and the granular ingredient which is not inflammability is said. As an example of said high specific-surface-area ingredient, there is an activated alumina, a silica, a zirconia, a titania, or a thing containing at least two of kinds of these. An activated alumina is desirable also among these in respect of high specific-surface-area organization potency, catalyst metal support ability, etc. As an activated alumina, there are gamma-alumina, beta-alumina, theta alumina, delta alumina, eta alumina, kappa alumina, etc. In order to make this high specific-surface-area ingredient adhere to the interior of the front face of the cel side attachment wall of said porosity ceramic honeycomb structure object, and pore firmly, it is desirable to mix with a binder and to use.

As an example of this binder, there are alumina sol, a silica sol, an aluminium nitrate, etc.

[0013] In the 2nd mode of this invention, the reason the particle diameter of high specific-surface-area ingredients, such as an activated alumina, is the above range is because a high specific-surface-area ingredient needs to trespass upon the interior of the pore of the cel side attachment wall of the porosity ceramic structure before coating by said high specific-surface-area ingredient. Although only the front face of a cel side attachment wall coated honeycomb mold monolith support with the high specific-surface-area ingredient conventionally, since the particulate contained in exhaust gas stops on the front face of the cel side attachment wall of a filter, and at the interior of the pore of a cel side attachment wall in the case of the honeycomb mold filter of the structure where exhaust gas passes through the interior of pore of a cel side attachment wall, since it burns in response to a catalysis, a particulate needs to contact inside this high specific-surface-area ingredient and pore at this time. Therefore, the aforementioned particle size is required for a high specific-surface-area ingredient.

[0014] As said inflammable destruction-by-fire matter, there are carbon, wheat flour, bread crumbs, corks, coal, tree waste, etc. The mean diameter of this inflammable destruction-by-fire matter is smaller than the average pole diameter of the porosity ceramic honeycomb structure object before coating by said high specific-surface-area ingredient. The reason is infiltrating into the interior of the pore of the cel side attachment wall of a filter with a high specific-surface-area ingredient, and after it is burned down, it is for the clearance made in the interior of pore protecting lock out of the pore by coating of a high specific-surface-area ingredient. It is not necessary to make particle size of the inflammable destruction-by-fire matter not necessarily the same as that of a high specific-surface-area ingredient. Since preferably sets the average pole diameter of the cel side attachment wall of the honeycomb structure object after, coating and calcinating said porosity ceramic honeycomb structure object with a high specific-surface-area ingredient on the other hand to 5 micrometers or more, the mean particle diameter of this inflammable destruction-by-fire matter is desirable for being referred to as 3 micrometers or more. Since the viscosity of a slurry becomes it high that this is less than 3 micrometers and it is hard coming to coat, it is not desirable.

[0015] The purpose which uses the inflammable destruction-by-fire matter is for controlling the pressure drop buildup of the filter by coating of a high specific-surface-area ingredient by making it distribute highly and carrying out coating of the high specific-surface-area ingredients, such as an activated alumina, to the interior of the pore of making the coating layer of a cel side-attachment-wall front face porosity-ize, and a cel side attachment wall. the case where carbon is used as inflammable destruction-by-fire matter -- a high specific-surface-area ingredient -- receiving -- at least -- more than 5wt% -- if it adds, effectiveness is to reduce a pressure loss. on the other hand -- carbon -- 50wt(s)% -- since bond strength with the filter of the coating layer of a high specific-surface-area ingredient will fall if it super-adds, it is not desirable.

[0016] There are an approach of coating with this invention using the solution which mixed the inflammable destruction-by-fire matter with the high specific-surface-area ingredient, and the approach of coating with the solution which contains only the inflammable destruction-by-fire matter beforehand, and coating with the solution containing the following high specific-surface-area ingredient. in order that the inflammable destruction-by-fire matter may coat with a high specific-surface-area ingredient the condition of having blockaded partially the interior of pore of the cel side attachment wall of a filter in the case of the latter -- porosity-izing of the coating layer inside pore -- and it can high-decentralize.

[0017] When coating with the solution which contains only the inflammable destruction-by-fire matter beforehand, as for the rate that the inflammable destruction-by-fire matter occupies in this solution, less than [ more than 5wt%50wt% ] is desirable. In the case of beyond 50wt%, since bond strength with the filter of the coating layer of a high specific-surface-area ingredient falls, it is not desirable.

[0018] The diesel emission-gas-purification filter of this invention carries out uptake of the particulate contained in the exhaust gas of a diesel power plant at least, and carries out combustion removal. A filter is coated with high specific-surface-area matter, such as an activated alumina, for making a platinum group catalyst metal into the support for carrying out coating. Generally, a platinum group catalyst metal is used as a catalyst which lowers a particulate combustion temperature, and is further used as an oxidation catalyst of a carbon monoxide or a hydrocarbon. The filter of this invention is a filter which has supported the metal catalyst which consists of at least one kind of platinum group metals. As said platinum group metals, at least one sort of platinum, a rhodium, palladium, a ruthenium, and iridium can be used.

[0019] Next, the diesel emission-gas-purification filter of this invention is concretely explained with drawing 1 and drawing 2 . It is the partial enlarged drawing of the A section [ in / drawing 1 , and in drawing 2 / drawing 1 ]. [ the sectional view of this invention diesel emission-gas-purification filter ] For the porosity ceramic filter of this honeycomb structure, the both ends of a monolith honeycomb are \*\*\*\*

suggestion \*\*\*\*\* by turns at the \*\*\*\*\* material 1. The coating layer 3 of an activated alumina is formed in the front face 21 of the cell wall of a honeycomb mold filter, and the pore front face 22 of a cell wall. Coating of the activated alumina is carried out to all cell walls, and it is raising the purification effectiveness of the exhaust gas components (HC, CO, etc.) of a particulate and others by which uptake was carried out inside the cell wall by supporting a platinum group catalyst metal into the coating part of an activated alumina. The publication of a platinum group catalyst metal is omitted in this drawing.

[0020] The diesel exhaust gas containing a particulate advances into a cel from the cel entrance side 4, passes a cell wall 2, and leaves it from the cel outlet side 5. At this time, uptake of the particulate is carried out by the pore of a cell wall front face and the interior. Although coating anew is common after a platinum group catalyst metal coats an activated alumina, it is also possible to coat with the solution mixed with an activated alumina and carbon.

[0021] The filter coated using the above ingredients can be suitably used as a diesel particulate filter of low voltage loss. Below, the example and example of a comparison are shown.

[0022]

[Example]

(Example 1) Produce the ceramic honeycomb structure object of a cordierite ( $2\text{MgO}$ ,  $2\text{aluminum}_2\text{O}_3$ , and  $5\text{SiO}_2$ ) presentation by the well-known extrusion process. It calcinates by the 1350 degrees C - 1450 degrees C maximum temperature, the programming rate of 5 degrees C - 200 degrees C, and the holding time of 2 - 20 hours. Porosity had the pore property which is the average pole diameter of 25 micrometers 55%, and the porosity cordierite honeycomb structure object with a diameter [ of 140mm ] and a die length of 130mm 0.45mm in cell wall thickness and whose number of cels per 1 square inch are 150 pieces was acquired.

[0023] On the other hand, as a high specific-surface-area ingredient, 1330g (gamma-alumina) (Sumitomo Chemical make) of activated aluminas of 5 micrometers of diameters of a centriole, Mix and agitate alumina sol (product made from Nissan chemistry) 670g with water 4L, and an activated-alumina slurry is produced. Six kinds of samples (carbon addition rate 10wt% - 100wt%) as for which carbon (product made from SEC) of 5 micrometers of diameters of a centriole changed the addition rate to this on the basis of the weight of an activated alumina were produced.

[0024] The aforementioned porosity cordierite honeycomb filter is completely dipped in said activated-alumina slurry containing carbon (wash coat). Then, the slurry which adhered too much in an air cleaner and compression Ayr is removed as completely as possible. It dried at 120 degrees C for 2 hours, and calcinated at 800 degrees C, and carbon was made furthermore burned down completely after that. Filter weight was measured after baking and the amount (g/L) of coats of per 1L was calculated from the weight difference with the filter in front of a wash coat.

[0025] Cel opening by the side of the gas inlet of the honeycomb structure object which carried out wash coat processing is \*\*\*\*\* (ed) every piece, and it \*\*\*\* [ chisel ] in a gas outlet side about the cel which is not \*\*\*\*\* (ing) by the entrance side. Especially as long as \*\*\*\*\* material is a ceramic ingredient with the thermal resistance of 1000 degrees C or more, such as cordierite, an alumina, and a zirconia, it may not be limited, but the adhesives made from a ceramic are sufficient as it. Thus, the filter with catalyst support was produced (Support A - support F).

[0026] (Example 2) The porosity cordierite honeycomb filter used for Example 1 and the same filter were produced, and the wash coat of said filter was carried out to the activated-alumina slurry which mixed and agitated 1330g of activated aluminas of 5 micrometers of diameters of a centriole, and alumina sol 670g with water 4L as a high specific-surface-area ingredient. Then, the slurry which adhered too much in an air cleaner and compression Ayr is removed as completely as possible. Furthermore, after that, it dried at 120 degrees C for 2 hours, and calcinated at 800 degrees C. Filter weight was measured after baking and the amount (g/L) of coats of per 1L was calculated from the weight difference with the filter in front of a wash coat.

[0027] Then, cel opening by the side of the gas inlet of the honeycomb structure object which carried out wash coat processing was \*\*\*\*\* (ed) every piece, in the gas outlet side, it \*\*\*\* (ed) [ chisel ] about the cel which is not \*\*\*\*\* (ing) by the entrance side, and the filter with catalyst support was produced (support G).

[0028] The porosity cordierite honeycomb filter used for Example 1 and the same filter are produced.

(Example 3) As a high specific-surface-area ingredient 1330g [ of activated aluminas of 5 micrometers of diameters of a centriole ] and alumina sol 670g is mixed with water 4L. The wash coat was carried out to the filter which produced six kinds of samples (carbon addition rate 10wt% - 100wt%) as for which carbon




(product made from SEC) of 50 micrometers of diameters of a centriole changed the addition rate to this on the basis of the weight of an activated alumina, and was produced by the same process as an example 1. Then, the slurry which adhered too much in an air cleaner and compression Ayr was removed as completely as possible. It dried at 120 degrees C for 2 hours, and calcinated at 800 degrees C, and carbon was made furthermore burned down completely after that. Filter weight was measured after baking and the amount (g/L) of coats of per 1L was calculated from the weight difference with the filter in front of a wash coat. [0029] Then, cel opening by the side of the gas inlet of the honeycomb structure object which carried out wash coat processing was \*\*\*\*\* (ed) every piece, in the gas outlet side, it \*\*\*\*\* (ed) [ chisel ] about the cel which is not \*\*\*\*\* (ing) by the entrance side, and the filter with catalyst support was produced (Support H - support M).

[0030] (Measurement of the pressure loss of a filter with catalyst support) The differential pressure of a sink, an entrance side, and an outlet side was measured for compression Ayr from the entrance side of a filter. The measurement result of the pressure loss of the filter with catalyst support obtained by Examples 1-3 is shown in Table 1 and drawing 3. From this result, the filter of Example 1 is understood that a pressure loss is lower than the filter of Examples 2 and 3.

[0031]

[Table 1]

担 体 の 種 類	A	B	C	D	E	F	G	H	I	J	K	L	M
カーボン粒径 (μm)	5	5	5	5	5	5		50	50	50	50	50	50
カーボン量 (g/活性アルミナ量100g)	10	20	30	50	70	100	0	10	20	30	50	70	100
活性アルミナの 担持量 (g/L)	61	60	65	68	70	70	65	62	66	61	60	63	66
圧力損失 (mmAq)	25	23.5	24	23	22	22.5	34	35	34	35	33	34	33

[0032] (Pore distribution measurement before and behind coating) The result of the pore distribution measurement before and behind coating of example 1 (support A) example 3 (support H) is shown in drawing 4 and drawing 5, respectively. In this invention article, even if it coats an activated alumina, the average pole diameter of pore of those with 5 micrometers or more and the blockaded cell wall of a filter has decreased. On the other hand, in Example 3, the average pole diameter was set to less than 5 micrometers by coating, and the pore blockaded has increased. That is, if carbon is made to trespass upon the interior of the pore of a filter, the interior of pore will not be buried with an activated alumina, and a pressure loss will become low.

[0033]

[Effect of the Invention] The diesel emission-gas-purification filter which has the porosity ceramic filter in which coating was carried out by this invention with the high specific-surface-area ingredient in the front face of a honeycomb structure object side attachment wall and the interior of pore is offered.

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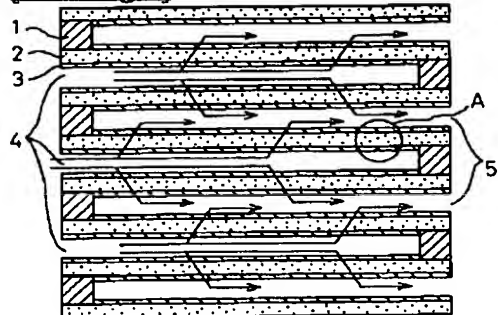
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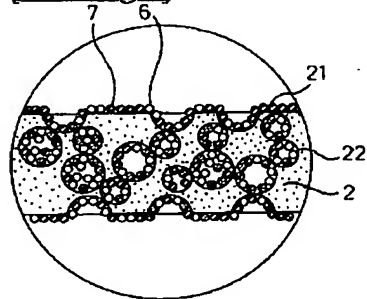
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## DRAWINGS

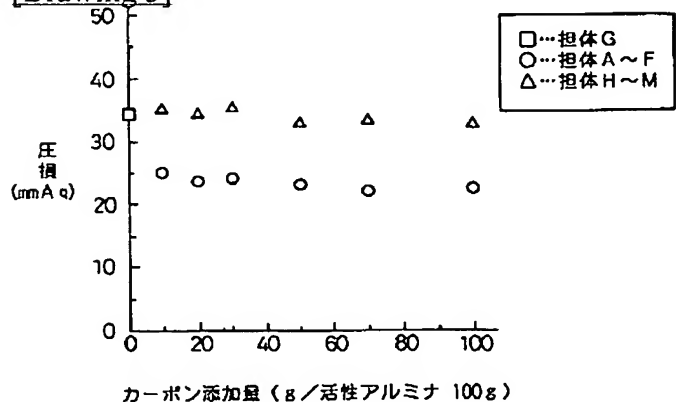
[Drawing 1]



[Drawing 2]



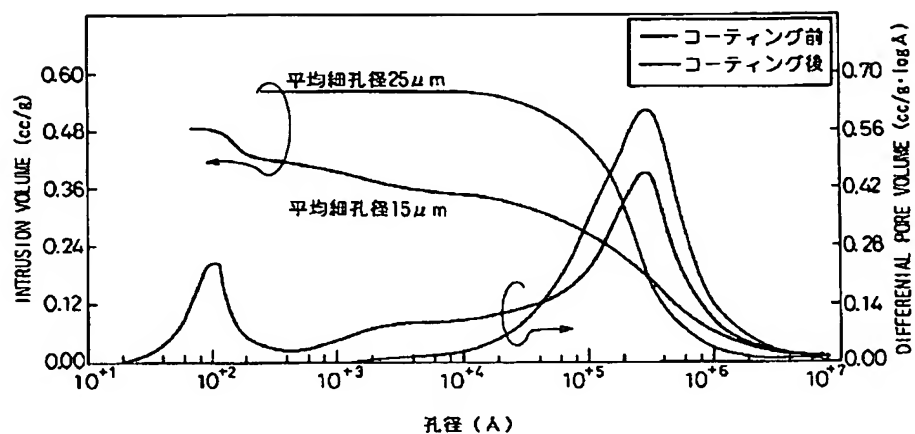
[Drawing 3]



[Drawing 4]

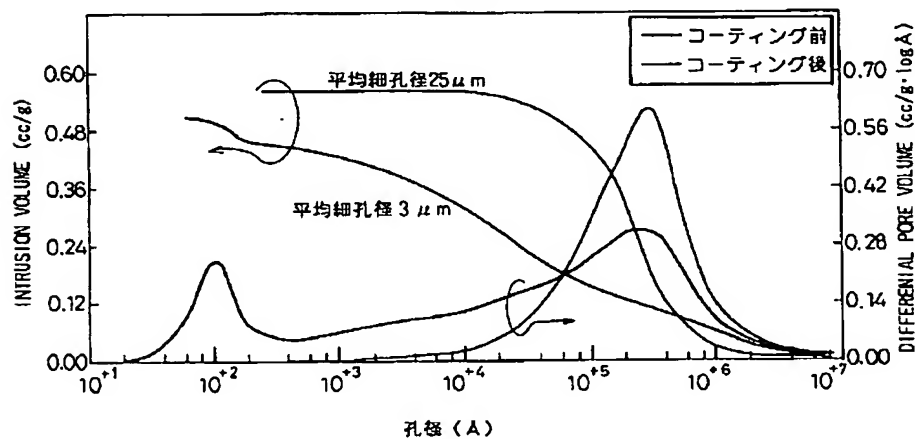


例 1



[Drawing 5]

例 3



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